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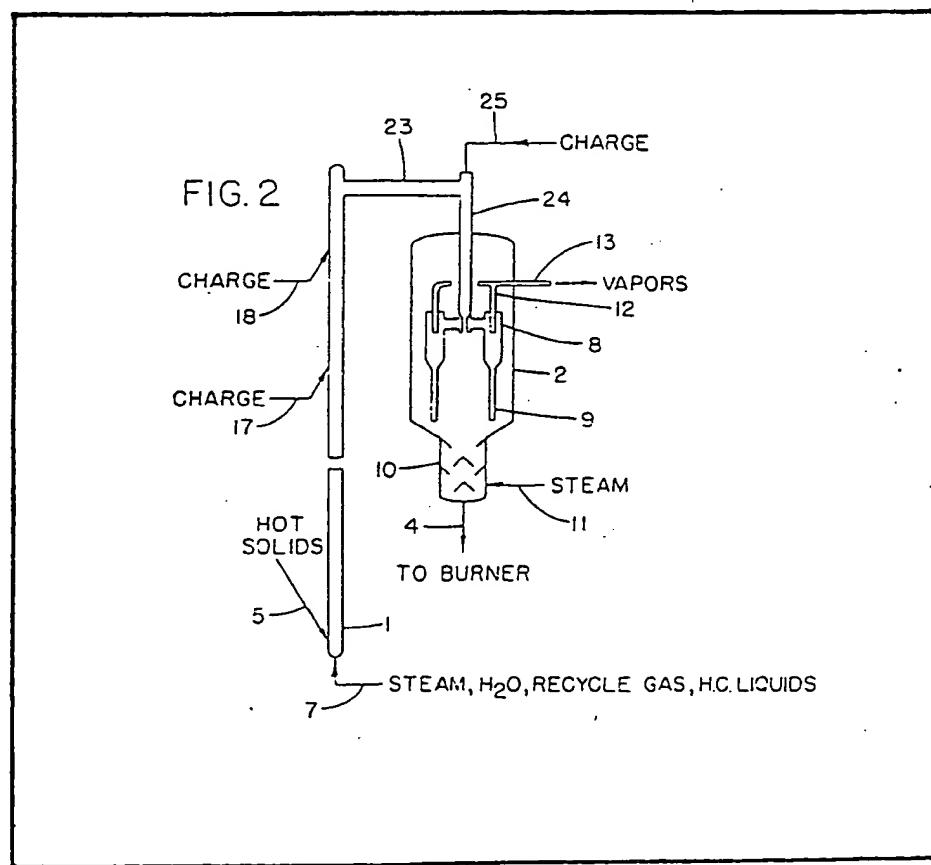
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(54) Decarbonizing and  
demetallizing petroleum stocks

(57) Heavy petroleum is contacted  
with a rising confined column 1 of hot  
inert solid (e.g. kaolin microspheres)  
and inert gas 7 (e.g. steam or  
methane) at a temperature and for a time  
such that vaporous products 13 are

obtained without substantial cracking.  
The inert solid 4 is separated from the  
vaporous product and contacted with  
an oxidizing gas to burn off  
combustible deposit and reheat the  
solid for recycle to the rising column.  
The petroleum can be injected into the  
column at one or more points 17, 18  
at or downstream of the injection of  
inert gas.



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FIG. I

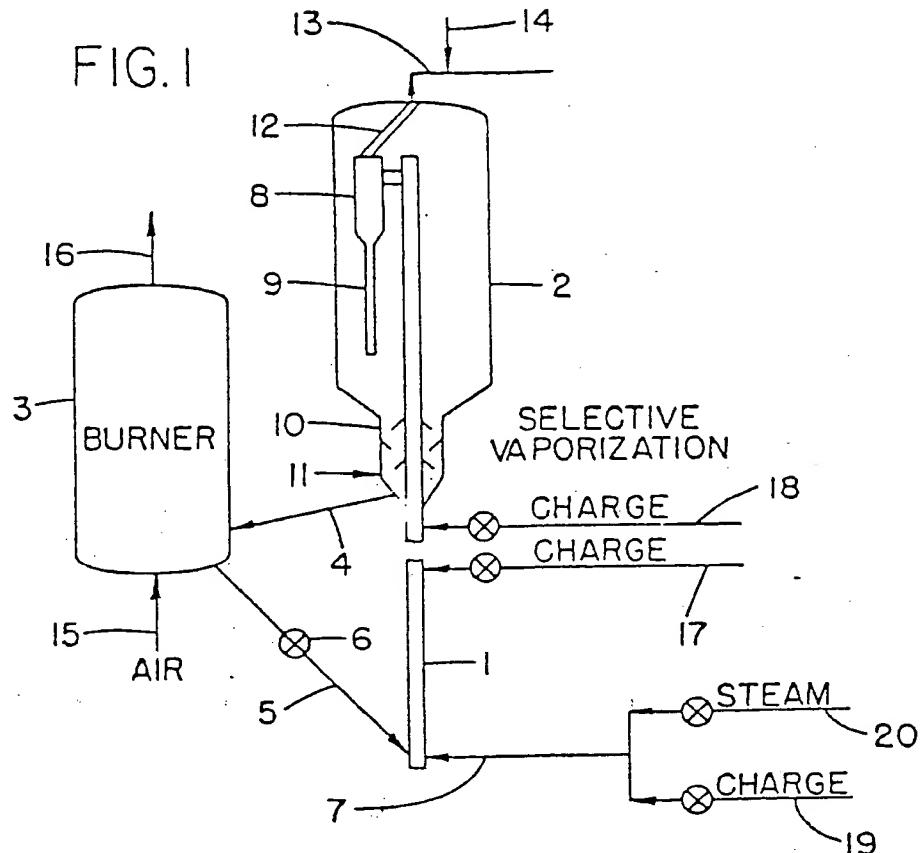
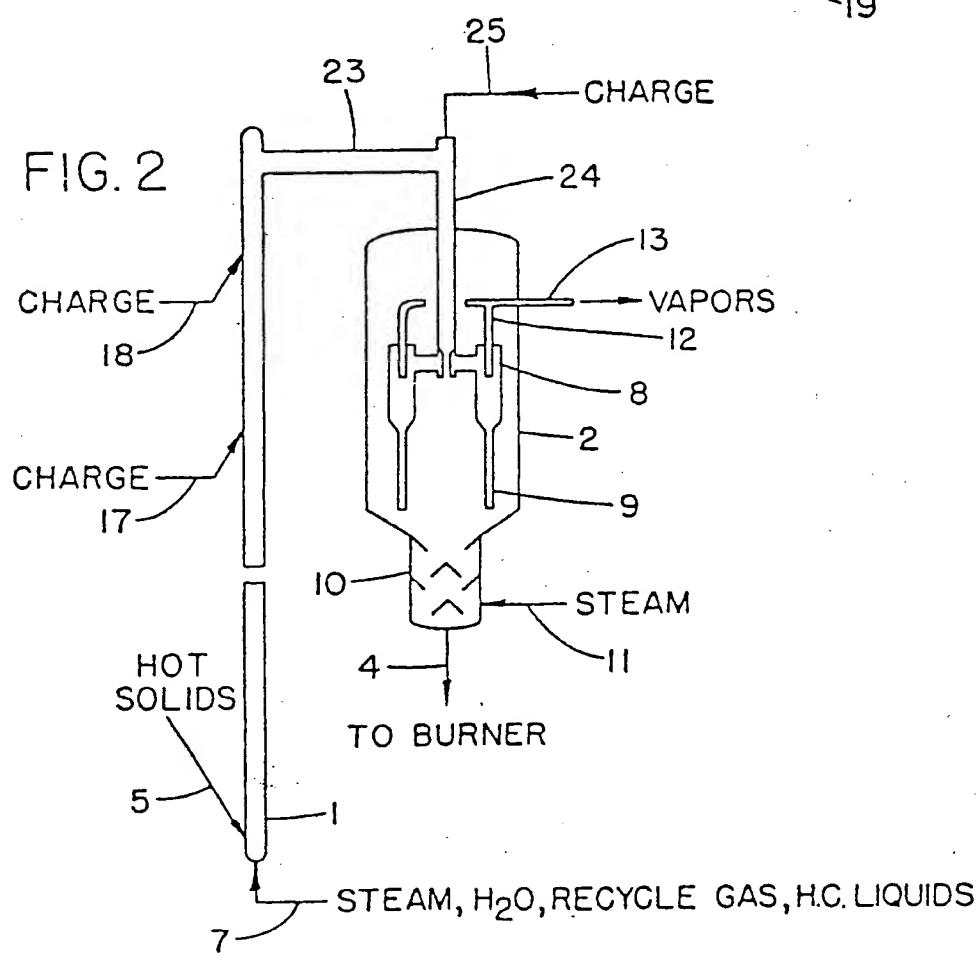


FIG. 2



## SPECIFICATION

Selective hydrocarbon vaporization process  
and apparatus

The invention is concerned with increasing the portion of heavy petroleum crudes which can be utilized as catalytic cracking feed stock to produce premium petroleum products, particularly motor gasoline of high octane number, or as high quality heavy fuel. The heavy ends of many crudes are high in Conradson Carbon (sometimes reported as Ramsbottom Carbon) and metals which are undesirable in catalytic cracking feed stocks and in products such as heavy fuel. The present invention provides an economically attractive method for selectively removing and utilizing these undesirable components from whole crudes and from the residues of atmospheric and vacuum distillations, commonly called atmospheric and vacuum residua or "resids". The terms "residual stocks", "resids" and similar terminology will be used here in a somewhat broader sense than is usual to include any petroleum fraction remaining after fractional distillation to remove some more volatile components. In that sense "topped crude" remaining after distilling off gasoline and lighter is a resid. The undesirable CC (for Conradson Carbon) and metal bearing compounds present in the crude tend to be concentrated in the resids because most of them have low volatility. The terms "Conradson Carbon" and "Ramsbottom Carbon" have reference to the two most used tests for this undesirable constituent. Some difference in numerical values by the two tests may be found for the same sample, but generally the test results from either are indicative of the same characteristic.

The invention is an improvement on the selective vaporization process and apparatus. The present invention relates to a selective vaporization process for decarbonizing and demetallizing heavy petroleum fractions by contacting such fraction and an inert gas for reduction of hydrocarbon partial pressure with finely divided inert solid contact material at low cracking severity conditions of high temperature and short hydrocarbon residence time in a rising confined column, separating vaporous products of said contacting from said contact material bearing a combustible deposit of unvaporized high Conradson Carbon or high metal content constituents of said petroleum fraction, quenching said vaporous products to a temperature below that at which substantial thermal cracking occurs, contacting said separated contact material with an oxidizing gas to burn said combustible deposit and heat the contact material to high temperature and returning the so heated contact material to the lower portion of said confined column for renewed contact with said heavy petroleum fraction; and it provides flexibility in rate of charging said fraction or in control of said residence time, or both, by introducing such inert

gas to the lower portion of said confined column, and injecting such heavy petroleum fraction into said column at and/or downstream of said lower portion. The present invention also relates to an apparatus for selective vaporization of a heavy petroleum fraction comprising a riser contactor in the form of a vertical conduit, a hot solids standpipe connected to the bottom of said riser contactor for supply thereto of finely divided hot inert solid contact material, means to supply an inert carrier gas to the bottom of said riser contactor to suspend said solid contact material in a rising confined column of gases and contact material in said riser contactor, a plurality of injection means for the introduction of heavy petroleum fraction into said riser contactor spaced at different levels of said riser contactor, means at the top of said riser contactor for separation of vapors from solid contact material bearing a combustible deposit, a burner, means to transfer the so separated solid contact material to said burner, air inlet means to said burner for supply of air thereto for combustion of said combustible deposit whereby the temperature of said solid contact material is increased and means to transfer the so heated contact material from said burner to the said hot solids standpipe.

The selective vaporization process is conducted by contacting a heavy charge stock such as whole crudes, topped crudes, resids and the like with an inert, finely divided solid material in a confined vertical column under conditions to deposit heavy components of high CC and/or metal content on the solid and vaporize other components of the charge. This results from

temperatures high enough to cause the desired vaporization and very short hydrocarbon residence times to avoid substantial cracking. The operation is thus held to a low cracking severity to accomplish the desired purpose of separating vaporizable, more valuable components from those which are regarded as contaminants. Steam, light hydrocarbons or the like are added to the rising confined column in the selective vaporization facility to reduce partial pressure of hydrocarbons in the charge and thus aid in vaporization.

Vaporous hydrocarbons are separated at the top of the column from inert solids bearing the unvaporized components as a deposit thereon. The vapors are promptly cooled to a temperature below that at which substantial thermal cracking occurs and processed as desired in a catalytic cracker or the like.

According to certain embodiments of the invention, the contact is conducted in a riser. In other embodiments, a rising column of inert solids in steam, hydrocarbon gases or both is established and the direction of flow is reversed to a confined descending column into which the charge is injected.

The separated inert solids bearing the deposit of unvaporized components of the charge are transferred to a burner for combustion of the deposit in air or other oxygen containing gas. Heat

generated by combustion of the deposit raises the temperature of the inert solids which are then returned to the lower portion of the rising confined column to supply the heat for selective vaporization of additional heavy charge.

5 The present invention provides a process and an apparatus for varying the hydrocarbon residence time in the riser which defines the confined rising vertical column in which selective vaporization is conducted. That result is 10 accomplished by providing a plurality of points for injection of charge stock to the selective vaporization riser to compensate for changes in quantity or quality of feed stock.

15 Apparatus suited to practice of the invention is illustrated diagrammatically in Figure 1 of the annexed drawings. Figure 2 represents an embodiment in which charge is injected to a descending column produced by reversing 20 direction of flow of a stream of suspended inert solids which is initially established as a rising column.

As shown in Figure 1, the principal vessels used are a riser 1 for conducting the short time, 25 high temperature contact between hot inert solids and charge stock which terminates in a disengaging chamber 2 from which inert solids bearing a deposit of unvaporized material are transferred to a burner 3 by standpipe 4. The hot 30 inert solids resulting from combustion in burner 3 are returned to the base of riser 1 by a standpipe 5 through a control valve 6.

A charge stock containing high boiling components which are characterized by high CC 35 or metals content or both is admitted to the riser 1 by line 7 to rise at high velocity in riser 1 while in intimate contact with the hot inert solids from standpipe 5. The major portion of the charge stock is vaporized at the temperature prevailing in 40 the riser by reason of the hot solids from standpipe 5. That vaporization is extremely rapid to result in a rapidly rising column of vapor with inert solids suspended therein. The portions of the charge which are not vaporized coalesce on the 45 inert solids to provide a combustible deposit constituted primarily by feed stock components of high CC and metals content.

The solids are separated from the vaporous hydrocarbons at the top of riser 1 by any of the 50 systems developed for the same purpose in the well-known FCC process for riser cracking of hydrocarbons in the presence of an active cracking catalyst. A system of preference in the present invention is the vented riser described in 55 U.S. patents 4,066,533 and 4,070,159. The upper end of riser 1 is open whereby inertia of the suspended solids causes them to be projected into the vessel 2. Vapors leave the riser through a side vent in the riser to cyclone separator 8 where 60 solids still suspended in the vapors are removed and discharged by dip leg 9 to the lower portion of vessel 2. The solids projected from the top of riser 2 and those from dip leg 9 pass downwardly to a stripper 10 where steam from line 11 aids in 65 vaporization of any remaining volatile

hydrocarbons before the solids bearing combustible deposit enter standpipe 4 for transfer to burner 3.

The vapors separated from entrained solids in 70 cyclone 8 pass by conduit 12 to transfer line 13 where the vapors are cooled to a temperature below that at which substantial thermal cracking occurs as by mixture with a suitable quench medium such as a cold hydrocarbon stream or 75 water. The burner 3 may be any of the various structures developed for burning of combustible deposits from finely divided solids, for example, the regenerators for Fluid Cracking Catalyst. Air admitted to the burner 3 by line 15 provides the 80 oxygen for combustion of the deposit on the inert solid, resulting in gaseous products of combustion discharged by flue gas outlet 16. The burner 3 is preferably operated to maintain the temperature in the burner at the maximum value, usually 85 limited by metallurgy of the burner. This is accomplished by controlling temperature of the riser 1 to the minimum temperature which will provide the amount of fuel (as deposit on the inert solids) which will sustain the maximum 90 temperature of the burner. As is common in heat balanced FCC Units, valve 6 is controlled responsive to the temperature at the top of riser 1 in a manner to maintain the riser temperature at a preset value. That preset temperature is reset as 95 needed in selective vaporization to maintain a desired maximum temperature in burner 3. A trend to lower temperature in burner 3 is compensated by reduction of the preset temperature of riser 1, and vice versa. Inert solids 100 heated by the combustion in burner 3 are stripped with steam in the burner 3 or the standpipe 5 before being returned to riser 1.

The solid contacting agent is essentially inert in the sense that it induces minimal cracking of 105 heavy hydrocarbons by the standard microactivity test conducted by measurement of amount of gas oil converted to gas, gasoline and coke by contact with the solid in a fixed bed. Charge in that test is 0.8 grams of mid-Continent 110 gas oil of 27 API contacted with 4 grams of catalyst during 48 second oil delivery time at 910°F. This results in a catalyst to oil ratio of 5 at weight hourly space velocity (WHSV) of 15. By that test, the solid here employed exhibits a 115 microactivity less than 20, preferably about 10. A preferred solid is microspheres of calcined kaolin clay. Other suitable inert solids include, in general any solid which satisfies the stated criteria.

120 The microspheres of calcined kaolin clay preferably used in the process of the invention are known in the art and are employed as a chemical reactant with a sodium hydroxide solution in the manufacture of fluid zeolitic cracking catalysts as 125 described in U.S. 3,647,718 to Haden et al. In practice of the instant invention, in contrast, the microspheres of calcined kaolin clay are not used as a chemical reactant. Thus the chemical composition of the microspheres of calcined clay 130 used in practice of this invention corresponds to

that of a dehydrated kaolin clay. Typically, the calcined microspheres analyze about 51% to 53% (wt.)  $\text{SiO}_2$ , 41 to 45%  $\text{Al}_2\text{O}_3$ , and from 0 to 1%  $\text{H}_2\text{O}$ , the balance being minor amounts of 5 indigenous impurities, notably iron, titanium and alkaline earth metals. Generally, iron content (expressed as  $\text{Fe}_2\text{O}_3$ ) is about 1/2% by weight and titanium (expressed as  $\text{TiO}_2$ ) is approximately 2%.

The microspheres are preferably produced by 10 spray drying an aqueous suspension of kaolin clay. The term "kaolin clay" as used herein embraces clays, the predominating mineral constituent of which kaolinite, halloysite, nacrite, dickite, 15 anauxite and mixtures thereof. Preferably, a fine particle size plastic hydrated clay, i.e., a clay containing a substantial amount of submicron size particles, is used in order to produce microspheres having adequate mechanical strength.

20 While it is preferable in some cases to calcine the microspheres at temperatures in the range of about 1600°F. to 2100°F. in order to produce particles of maximum hardness, it is possible to dehydrate the microspheres by calcination at 25 lower temperatures; for example, temperatures in the range of 1000°F. to 1600°F., thereby converting the clay into the material known as "metakaolin". After calcination the microspheres should be cooled and fractionated, if necessary, to 30 recover the portion which is in the desired size range, say 20—150 microns.

Pore volume of the microspheres will vary slightly with the calcination temperature and duration of calcination. Pore size distribution 35 analysis of a representative sample obtained with a Desorpta analyzer using nitrogen desorption indicates that most of the pores have diameters in the range of 150 to 600 Angstrom units.

The surface area of the calcined microspheres 40 is usually within the range of 10 to 15  $\text{m}^2/\text{g}$ . as measured by the well-known B.E.T. method using nitrogen absorption. It is noted that the surface areas of commercial fluid zeolitic catalysts is considerably higher, generally exceeding values of 45 100  $\text{m}^2/\text{g}$ . as measured by the B.E.T. method.

Although the system just described bears 50 superficial resemblance to an FCC Unit, its operation is very different from FCC. Most importantly, the riser contactor 1 is operated to remove from the charge an amount not greatly in 55 excess of the Conradson Carbon number of the feed. This contrasts with normal FCC "conversion" of 50—70%, measured as the percentage of FCC product not boiling within the range of the charge. Percent removed by the present process is preferably on the order of 10% to 20% on charge and constituted by gas, gasoline and deposit on the solid contacting agent. Rarely will the amount removed as gas, 60 gasoline and deposit on the inert solid exceed a value, by weight, more than 3 to 4 times the Conradson Carbon value of the charge. This result is achieved by a very low severity of cracking due to inert character of the solid and the very short 65 residence time at cracking temperature. As is well

known, cracking severity is a function of time and temperature. Increased temperature may be compensated by reduced residence time, and vice versa.

70 The new process affords a control aspect not available to FCC Units in the supply of hydrocarbons or steam to the riser contactor. When processing stocks of high CC, the burner temperature will tend to rise because of increased

75 supply of fuel to the burner. This may be compensated by increasing the hydrocarbons or steam supplied to reduce partial pressure of hydrocarbons in the riser contactor or by recycling water from the overhead receiver to be vaporized

80 in the riser to produce steam.

The riser contact with inert solid thus provides a novel sorption technique for removing the polynuclear aromatic compounds of resids (high CC and metals) while these are carried in a stream of

85 low hydrocarbon partial pressure by reason of hydrocarbons or steam supplied to the riser.

The decarbonized, desalts and/or demetallized resid is good quality FCC charge stock and may be transferred to the feed line of an

90 FCC reactor operated in the conventional manner.

It is found that the nature of the selective vaporization is a function of temperature, total pressure, partial pressure of hydrocarbon vapors, residence time, charge stock and the like. One 95 effect of temperature is a tendency to decrease the combustible deposit on the contact material as contact temperature is increased. Thus greater portions of the charge are vaporized at higher temperatures and the secondary effect of thermal

100 cracking of deposited hydrocarbons increases at higher temperatures. These effects of higher temperature enhance the yield of product from the operation and reduce the fuel supplied to the combustible deposit.

In general, the temperature of selective vaporization will be above the average boiling point of the charge stock, calculated as the sum of the 10% to 90% points by ASTM distillation of the

110 charge divided by nine. For the heavy stocks contemplated by the invention, the contact temperature will usually be not substantially below 900°F., and will be below the temperatures at which severe cracking occurs to produce large 115 yields of olefins. Thus even at residence times as short as 0.1 second or less, selective vaporization temperatures will be below about 1050°F.

Residence time for selective vaporization is not accurately calculated by the methods generally 120 used in FCC cracking where the volume of vapors increases to a major extent as the hydrocarbons remain in contact with an active cracking catalyst along the length of a riser. In selective vaporization, the vapors are quickly generated on

125 contact with the hot inert solid and remain substantially constant in composition along the length of the riser, increasing slightly with modest thermal cracking believed to be cracking of the deposit on the inert solid. Residence time of

130 hydrocarbons in selective vaporization is therefore

calculated with reasonable accuracy as the length of the riser from point of hydrocarbon injection to point of disengagement from inert solids divided by superficial velocity of vapors (hydrocarbons, 5 steam, etc.) at the top of the riser. So calculated, hydrocarbon residence time in selective vaporization will be not substantially greater than about 3 seconds and is preferably much shorter, one second or less, such as 0.1 second. As 10 previously indicated, residence time and temperature will be correlated to provide conditions of low cracking severity. The quantity removed from the charge is very nearly equal to CC value of the charge when operating under 15 preferred conditions and will rarely exceed a value 3 to 4 times the CC of the charge. Further, the hydrogen content of the deposit is about 3% to 6%; below the 7—8% normal in FCC coke.

The invention provides a means to vary 20 hydrocarbon residence time while maintaining charge rate constant or to maintain a constant residence time at reduced charge rate. It will be apparent that the invention also provides other flexibilities for the process, i.e., residence times 25 and/or charge rates may be varied without holding either at constant levels.

That effect results from use of a riser 1 which has multiple injection points along the length thereof. When hydrocarbon feed is injected at a 30 point above the bottom of the riser, an inert gas is injected at the bottom of the riser to carry the inert solids upwardly to the region of hydrocarbon injection. That inert gas also serves the function of reducing hydrocarbon partial pressure above 35 the point of hydrocarbon injection, thus promoting selective vaporization. The inert gas is preferably supplied as steam or water but may be any gas which will not undergo substantial reaction at the conditions prevailing in the riser. 40 Thus the process may use nitrogen as the lift gas or may use a hydrocarbon which will not undergo substantial thermal cracking at the riser conditions. Methane and other light hydrocarbons which boil below about 450°F. are preferred 45 examples of such materials.

As shown in Figure 1, the riser 1 is provided with injection means to supply charge from valved lines 17 and 18 which may be conveniently spaced at 25% and 50% of the height 50 of riser 1, respectively. In a riser so modified, injection line 7 at the bottom of the riser is provided with valved lines 19 and 20 for supply of steam or other recycle materials such as sour water, recycle gas or hydrocarbon liquids to the 55 bottom of riser 1 with or without hydrocarbon charge stock.

As stated above, water may be admitted to the bottom of riser 1 from line 7 to provide the lift gas in the lower portion of riser 1 and to reduce 60 hydrocarbon partial pressure above the points of charge injection. It will be understood that the term "water" is used here as indicating the liquid phase, as distinguished from steam. Supply of liquid water to the bottom of the riser 1 results in 65 immediate vaporization of the water in contact

with hot inert solids from standpipe 5 providing steam for the purpose stated. For reasons which are not presently understood, injection of liquid water to the bottom of the riser 1 results in 70 increased hydrogen content of the vapor product taken off at line 13 as compared with steam injection to the bottom of the riser. It has been found that addition of sulfur bearing compounds such as hydrogen sulfide, mercaptans and the like 75 suppresses that hydrogen generation tendency of liquid water injection.

It will be recognized that injection of liquid water to the bottom of the riser will reduce temperature of the inert solid to an extent. 80 corresponding to latent heat of vaporization plus the heat required to raise the temperature of the resultant water vapor. That will be compensated by automatic increase in supply of hot inert solid to the bottom of the riser and increase in the ratio 85 of inert solid to hydrocarbon charge in the riser.

It is also contemplated that steam, water, recycle gas and hydrocarbon liquid could be added with the hydrocarbon charge stock in the upper injection points or added after the 90 hydrocarbon charge stock. The advantage of adding recycle materials such as steam, liquid water, recycle gas or hydrocarbon liquids after the charge is to increase the rate of inert solid circulation and thereby increase the charge-inert 95 solid contact temperature. This will aid in the selective vaporization of the heavier hydrocarbons in the charge and still result in low contact times.

In the embodiment of Figure 2, the flow in riser contactor 1 is reversed to enter the vessel 2 by 100 downward flow through the top of vessel 1. Various structures for that purpose have been designed for parallel use in the FCC process, as generally discussed in column 4, lines 42—59 of the above-cited U.S. Patent 4,070,159. As shown 105 in Figure 2, a column of rising hot inert solids is established in riser contactor 1 by injecting steam, liquid water, recycle gas or stable light hydrocarbon liquid to the bottom of the riser by line 7 to mix with and suspend hot inert solids 110 added by standpipe 5 from the burner. Charge residual fractions or recycle such as steam, liquid water, recycle gas or hydrocarbon liquid may be added at points along riser 1 from line 17 and 18 as in Figure 1. At the highest point of riser 1, flow 115 is directed horizontally through portion 23 of the contactor and then downwardly through vertical section 24 to the open end of the contactor with diversion of vapors to cyclones 8.

Significant advantages are realizable by adding 120 some or all of the charge to the upper end of contactor section 24. Extremely short contact times characterize this embodiment. In addition, the force of gravity on the inert solid does not induce the "slippage" which causes the inert solid 125 to have a longer residence time in the contactor than does the hydrocarbon vapor generated by contact of charge stock with hot inert solids in the embodiment of Figure 1. With some types of residual fractions, it will be found that best results 130 are achieved by injecting the total charge by line

25 to the downwardly directed section 24 of the contactor 1. In that type of operation, the riser portion of the contactor serves to establish the suspension of hot inert solids on the gaseous 5 medium which also acts to reduce partial pressure of hydrocarbon vapors produced by contacting the residual fraction charge with hot inert solids.

This system allows the operator increased 10 flexibility in the conduct of selective vaporization. Taken with the flexibility inherent in ability to vary the ratio between charge and steam, a unit can be operated over a wide range of charge stocks and residence time to adapt the operation to changes 15 in quantity and/or quality of charge available.

The invention thus provides a selective 20 vaporization method and apparatus using hot riser contacting of heavy petroleum fraction charge with inert gas and hot fine solid inert contact 25 material, in which such gas and contact material are introduced at the beginning of the riser and such charge can be introduced at the beginning of the riser and/or downstream thereof.

#### Claims

25 1. A selective vaporization process for decarbonizing and demetallizing heavy petroleum fractions comprising contacting such fraction and inert gas in a rising confined column with hot finely divided inert solid contact material at a 30 temperature and for a hydrocarbon residence time such as to yield vaporous products without substantial cracking, separating vaporous products of said contacting from said contact material now bearing a combustible deposit of 35 unvaporized high Conradson Carbon or high metal content constituents of said petroleum fraction, quenching said vaporous products to a temperature below that at which substantial thermal cracking occurs, contacting said 40 separated contact material with an oxidizing gas to burn said combustible deposit and heat the contact material to high temperature, and returning the so heated contact material to the lower portion of said confined column for 45 renewed contact with further heavy petroleum fraction, inert gas being introduced to a lower portion of said confined column and heavy petroleum fraction being injected into said column at and/or downstream of said lower 50 portion.

2. A process according to claim 1 wherein said inert gas comprises steam.

3. A process according to claim 2 wherein liquid water is introduced to the lower portion of 55 said confined column to generate steam as inert gas.

4. A process according to claim 3 wherein sulfur bearing compound is also introduced to the lower portion of said column.

5. A process according to claim 1 or 2 or 3 wherein said inert gas comprises hydrocarbon.

6. A process according to any preceding claim wherein said heavy petroleum fraction comprises a residual fraction.

65 7. A process according to any preceding claim wherein at least one of steam, water, recycle gas and hydrocarbon liquid is introduced into said column with said heavy petroleum fraction.

8. A process according to any preceding claim 70 wherein at least one of steam, water, recycle gas and hydrocarbon liquid is introduced into said column after said heavy petroleum fraction.

9. A process according to any preceding claim wherein a rising column of contact material 75 suspended in inert gas is established in said lower portion and heavy hydrocarbon is injected into the pre-established column downstream of said lower portion.

10. A process according to any of claims 1 to 8 80 wherein the direction of flow of said confined column is reversed and discharged as a downwardly flowing stream into an enlarged separation zone for conduct of the separation of said vaporous products from said contact material.

11. A process according to claim 10 wherein heavy petroleum fraction is injected into said downwardly flowing stream.

12. A process according to any preceding 90 claim wherein the position of injection is varied to vary said residence time.

13. Apparatus for selective vaporization of a heavy petroleum fraction comprising a riser contactor in the form of a vertical conduit, a hot 95 solids standpipe connected to the bottom of said riser contactor for supply thereto of finely divided hot inert solid contact material, means to supply inert carrier gas to the bottom of said riser contactor to suspend said solid contact material 100 in a rising confined column in said riser contactor, a plurality of injection means for the introduction of heavy petroleum fraction to said riser contactor spaced at different levels of said riser contactor, means at the end of said riser contactor for 105 separation of vapors from solid contact material bearing a combustible deposit, a burner, means to transfer the so separated solid contact material to said burner, air inlet means to said burner for supply of air thereto for combustion of said 110 combustible deposit whereby the temperature of said solid contact material is increased, and means to transfer the so heated contact material from said burner to said hot solids standpipe.

14. Apparatus according to claim 13 wherein 115 the said riser contactor is formed with a reverse bend at the upper end thereof.

15. Apparatus for selective vaporization of a heavy petroleum fraction, the apparatus having a rising column contactor which feeds into a 120 separator for the separation of solids from vapours and which has a lower portion adapted to receive inert gas and hot fine solid contact material and a plurality of longitudinally spaced inlets for heavy petroleum fraction.

16. A selective vaporization process substantially as hereinbefore described with reference to Fig. 1 of the accompanying drawings.

17. A selective vaporization process

substantially as hereinbefore described with reference to Fig. 2 of the accompanying drawings.

18. Selective vaporization apparatus substantially as hereinbefore described with

5 reference to Fig. 1 of the accompanying drawings.

19. Selective vaporization apparatus

substantially as hereinbefore described with reference to Fig. 2 of the accompanying drawings.

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